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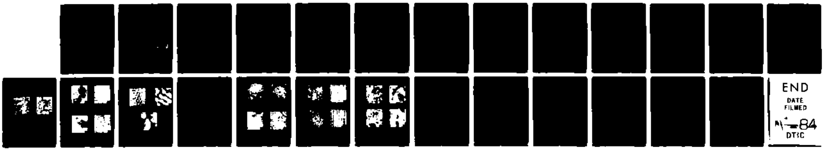

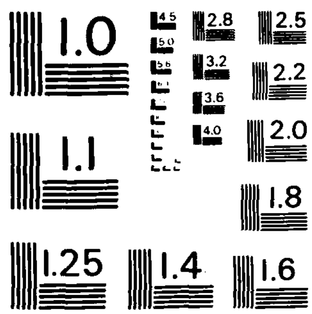
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ANNUAL TECHNICAL REPORT

# "A FUNDAMENTAL STUDY OF P/M PROCESSED ELEVATED TEMPERATURE ALUMINUM ALLOYS"

AFOSR ~~AFOSR Grant #~~ 82-0010

Principal Investigators: A. Lawley and M.J. Koczak

Department of Materials Engineering

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Philadelphia, Pa. 19104



March 1983

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Aluminum alloys exhibiting high strength and improved creep resistance at elevated temperatures offer the potential for lower weight and reduced cost in aerospace components. Powder processing, involving controlled atomization and hot consolidation, provides a means for fabricating candidate alloys; the microstructure consists of a stable fine-scale uniform dispersion of intermetallics in the aluminum matrix. Retention of elevated temperature strength has been demonstrated in a P/M Al-Fe-Ni alloy. Atomized powder is characterized

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20. ABSTRACT (continued)

by a duplex microstructure of fine and coarse regions of $FeNiAl_9$ ($V_f \approx 0.3$) in the aluminum matrix. The fine microstructure is harder than the coarse microstructure and is stable up to ~350°C, above which its hardness decreases rapidly. There is a gradual coarsening and decrease in hardness of the initially coarse regions with increasing temperature. The duplex microstructure is carried over into the hot pressed and extruded material. Changes in microstructure and hardness of the extruded material during elevated temperature exposure are similar to those occuring in the powder form. Hot tensile test data (up to 400°C) indicate that the extruded material retains ~60% of its ambient strength up to ~250°C with ductility approaching 10%. This reflects a promising level of structural stability. These results and observations can be explained in terms of particle cooling rate, precipitation of aluminides, and aluminide coarsening during powder processing; powder consolidation temperatures should be kept as low as possible.

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## ABSTRACT OF RESULTS

Aluminum alloys exhibiting high strength and improved creep resistance at elevated temperatures offer the potential for lower weight and reduced cost in aerospace components. Powder processing, involving controlled atomization and hot consolidation, provides a means for fabricating candidate alloys; the microstructure consists of a stable fine-scale uniform dispersion of intermetallics in the aluminum matrix. Retention of elevated temperature strength has been demonstrated in a P/M Al-Fe-Ni alloy. Atomized powder is characterized by a duplex microstructure of fine and coarse regions of $FeNiAl_9$ ($V_f \approx 0.3$) in the aluminum matrix. The fine microstructure is harder than the coarse microstructure and is stable up to ∿350°C, above which its hardness decreases rapidly. There is a gradual coarsening and decrease in hardness of the initially coarse regions with increasing temperature. The duplex microstructure is carried over into the hot pressed and extruded material. Changes in microstructure and hardness of the extruded material during elevated temperature exposure are similar to those occuring in the powder form. Hot tensile test data (up to 400°C) indicate that the extruded material retains ∿60% of its ambient strength up to ∿250°C with ductility approaching 10%. This reflects a promising level of structural stability. These results and observations can be explained in terms of particle cooling rate, precipitation of aluminides, and aluminide coarsening during powder processing; powder consolidation temperatures should be kept as low as possible.

## SUMMARY OF RESULTS

### a) Background

As a result of several recent studies, a viable basis now exists for the development of elevated temperature aluminum alloys for aerospace applications in the 230-350°C temperature range (1). Acceptable alloys must exhibit strength retention over this temperature range, which will necessarily require microstructural stability. With appropriate alloying additions, and utilizing powder metallurgy (P/M) processing science and technology, it is possible to achieve a high-volume fraction of stable, incoherent finely dispersed intermetallics in the matrix (2-5). These provide dispersion hardening and microstructural stability during elevated temperature exposure. In contrast, the slower solidification rates inherent in conventional casting preclude the development of such microstructures.

At the present time, only a limited understanding exists of composition, processing, microstructure, property and performance relationships in these P/M processed aluminum alloys. It is the overall objective of the present on-going study to develop such fundamental relationships. Attainment of optimum properties reflects a complex interplay of powder solidification rate, composition, mode(s) of consolidation and subsequent deformation processing. Properties being evaluated are elevated temperature strength and creep resistance, elastic modulus, ductility, toughness and fatigue response.

Several compositions in the Al-Fe-Ni system are included in this study; the system can be considered as a model dispersion-strengthened alloy in which the dispersoid is $FeNiAl_9$. Strength at 232°C is $\sim$7% lower than in the Al-Fe-Ce system but ductility is appreciably higher (2). Previous work on Al-Fe-Ni (6,7) has provided some insight into the

influence of powder particle morphology, particle size distribution and particle bonding integrity on strength, ductility and toughness. The present study complements a similar on-going AFOSR program by Fine and Weertman (8) on the Al-Fe-Ce system.

First year results and observations are summarized in this report and technological implications delineated. Studies have focussed on elevated-temperature microstructural stability and hot-tensile deformation.

b) Procedures

Atomized powder of composition Al-6.2 w/o Ni-5.9 w/o Fe (Al-3 a/o Ni-3 a/o Fe) was received from Alcoa. Powder surfaces and internal structure were characterized by means of SEM and optical microscopy, respectively. Dispersoid morphology was examined by TEM, using foils prepared from the powder by the technique of cold sintering (9). Powders were then exposed for 1 hour at temperatures up to 600°C to evaluate microstructural stability and microhardness.

Powder was hot pressed to full density at 371°C and subsequently extruded at the same temperature, using a 16:1 extrusion ratio. The hot pressed and extruded powder alloy was then exposed for 1 hour periods up to 600°C. After both modes of consolidation, the microstructure was examined optically and by TEM. Micro and macrohardness were measured as a function of prior elevated temperature exposure. Tensile tests were conducted at temperatures up to 400°C on the extruded material.

c) Powders

The air atomized powders are irregular in shape with rough surfaces, Figure 1. Average particle diameter is 11.4μm with 92.8% by weight of the powder <44μm. Internally, as-atomized powder particles are characterized by a duplex structure of fine (A) and coarse (B) regions, Figure 2; the

coarse structure is dendritic in appearance. After exposure at 400°C, some coarsening of both the fine and coarse regions of the structure is apparent, Figure 3. TEM of the powder more clearly illustrates coarsening of the $FeNiAl_9$ dispersoid above 400°C; examples for the fine structure are shown in Figure 4. The fine scale microstructure is harder than the coarse microstructure, as shown in Figure 5, and is stable up to ∿350°C. A gradual coarsening of the initially coarse regions takes place during elevated temperature exposure, with an accompanying small decrease in microhardness, Figure 5.

d) Consolidated Material

The duplex microstructure is carried over into the hot pressed and extruded material, and remains after elevated temperature exposure of both consolidated forms of the alloy. Representative microstructures of the hot-pressed material, before and after exposure at 400°C, are shown in Figure 6. Coarse and fine-scale regions (corresponding to the original powder particles) are clearly seen and some coarsening is evident after exposure at 400°C; this is generally more apparent in the finer than in the coarser particles. From TEM observations, it is seen that coarsening of the intermetallic occurs by a break-up and rounding of the lamellar form, Figure 7. The hardness of the hot-pressed material begins to drop significantly after exposure at temperature above ∿350°C.

Representative microstructure of extruded material in the transverse and longitudinal orientations are shown in Figure 8. It is seen that after hot pressing and extrusion, the original duplex microstructure present in the powder particles is retained. Elevated-temperature exposure at 400°C results in some coarsening of the intermetallic. Changes in the scale and morphology of the fine and coarse regions of the duplex microstructure in extruded material are clearly delineated by TEM, Figure 9.

Microhardness response of the fine and coarse regions in the extruded material to elevated-temperature exposure is similar to that exhibited by the alloy in powder form, cf. Figure 10 and Figure 5. Macrohardness of the extruded material is shown in Figure 11 as a function of prior exposure temperature.

Tensile properties of the extruded material, measured at temperature are given in Figure 12. Yield and tensile strength exhibit a similar temperature dependence. Above ~250°C there is a significant increase in ductility with a loss in strength. This is reflected in the appearance of fracture surfaces. Further high temperature tests are in progress. Concurrently, a detailed TEM study of the deformed material has been initiated in order to understand deformation mode(s), dislocation-particle interactions, and the effect of coarsening on tensile response. Fracture surface characterization (SEM) is in-progress to determine crack initiation and propagation details.

e) Interpretation and Significance of Results

As the size of the atomized liquid particles decreases, the degree of supercooling increases. Thus, the probability of a temperature rise in the particle above the solidus due to recalescence is low. This results in a fine-scale structure throughout the particle. For a larger diameter particle, the degree of undercooling is lowered and the probability of the recalescence temperature exceeding the solidus temperature is higher. Under this condition, the initial fine-scale structure will give way to a coarser structure in the remainder of the particle. The frequency of occurence of the duplex microstructure was observed to increase with increasing particle size. The powder being used in this study contains ~7% by weight above 44 μm dia.

The hardenss difference between the fine and coarse structures in the powder reflects a combination of a finer dispersion of intermetallics and a higher level of Fe and Ni in solid solution in the smaller particles. Hardness decreases in both the fine and coarse structural regions of the extruded material above ∿350°C are attributed to a gradual coarsening of the $FeNiAl_9$ intermetallic. The larger surface area and smaller inter-dispersoid spacing in the fine-scale regions account for a more pronounced change in the appearance of these regions than in the coarser regions.

Hot tensile test data indicate that the extruded material retains ∿60% of its ambient strength up to ∿250°C with ductility approaching 10%. This reflects a promising level of structural stability. In the as-extruded condition, ambient strength increases as the structure becomes finer; this should result in further enhancement in strength at elevated temperature. Key to finer structures resides in lower powder consolidation temperatures. To this end, samples of the Al-Fe-Ni powder were compacted by cold sintering (≡ high pressure compaction at ambient temperature) to essentially full-density at pressures > 3GPa (9). Prior to hot compressive testing, these compacts were annealed at 300°C and 371°C respectively; the latter corresponds to the extrusion temperature. Yield strength data are given in Figure 13. It is seen that cold sintering and annealing provides a strength increment relative to extruded material at elevated temperature; the effect is larger for the lower annealing temperature. Further studies utilizing the cold sintering approach to powder compaction are in-progress.

Overall, the study is designed to give a comprehensive and quantitative understanding of the interplay of composition, powder processing, microstructure and mechanical properties in the Al-Fe-Ni system. This will

enable reliable design guidelines to be established in terms of the definition of processing temperature ranges and elevated temperature application of these alloys. Further, because of the fundamental nature of the information gained in this program, it should be applicable to other aluminum-base high temperature systems. Composition as a variable will be reflected in different levels of dispersoid contents, above and below the level examined to-date (approximately 30 $^{v}/o$).

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9. E. Gutmanas, A. Lawley and M. Premkumar, "Microstructure and Mechanical Properties of Cold-Sintered P/M Aluminum Alloys", P/M '83 Conference Proceedings, APMI, Princeton, N.J., in press.

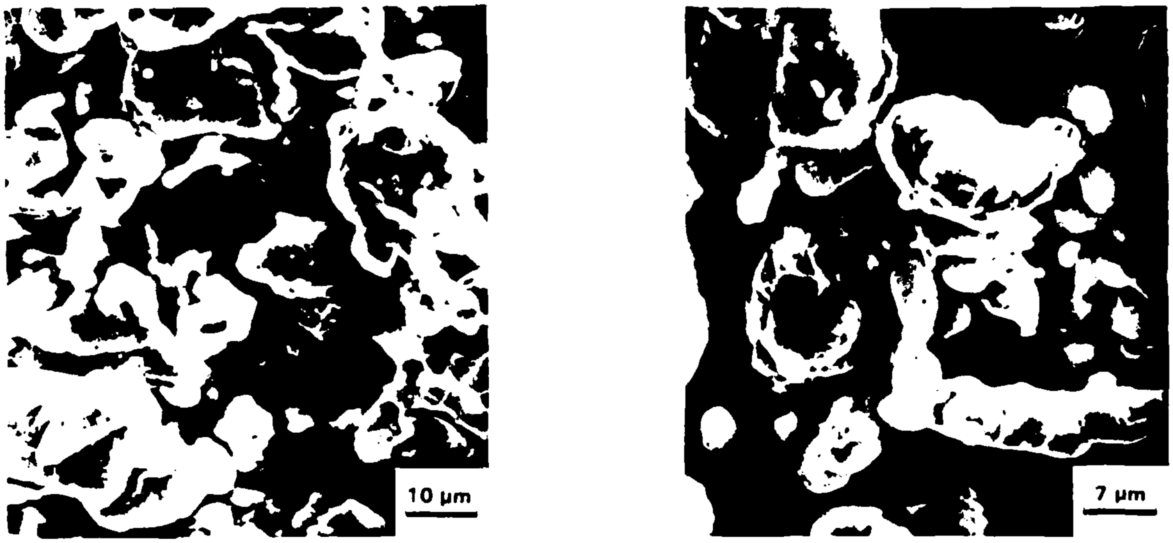
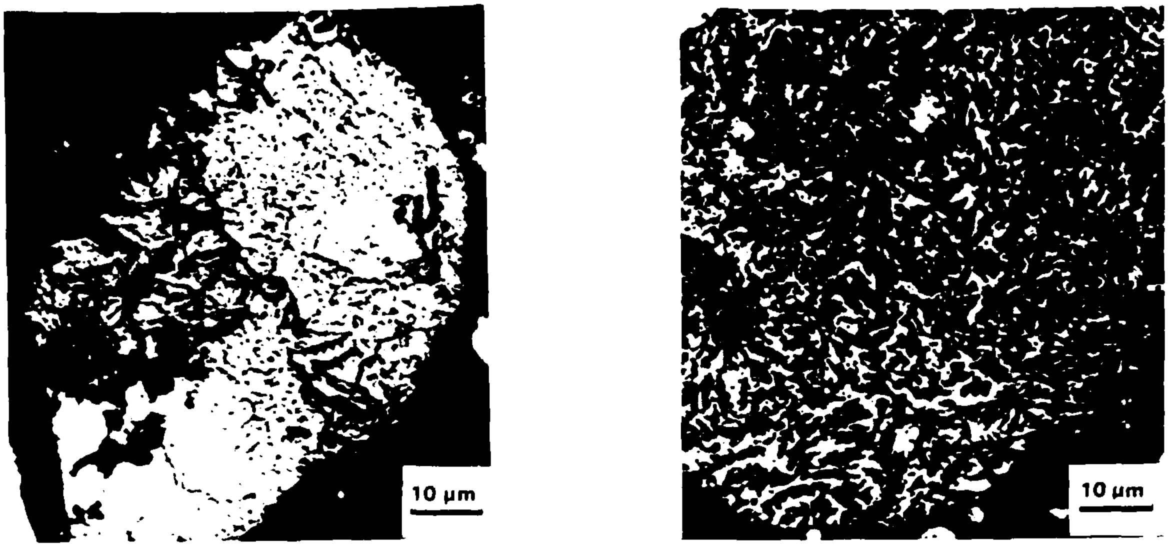
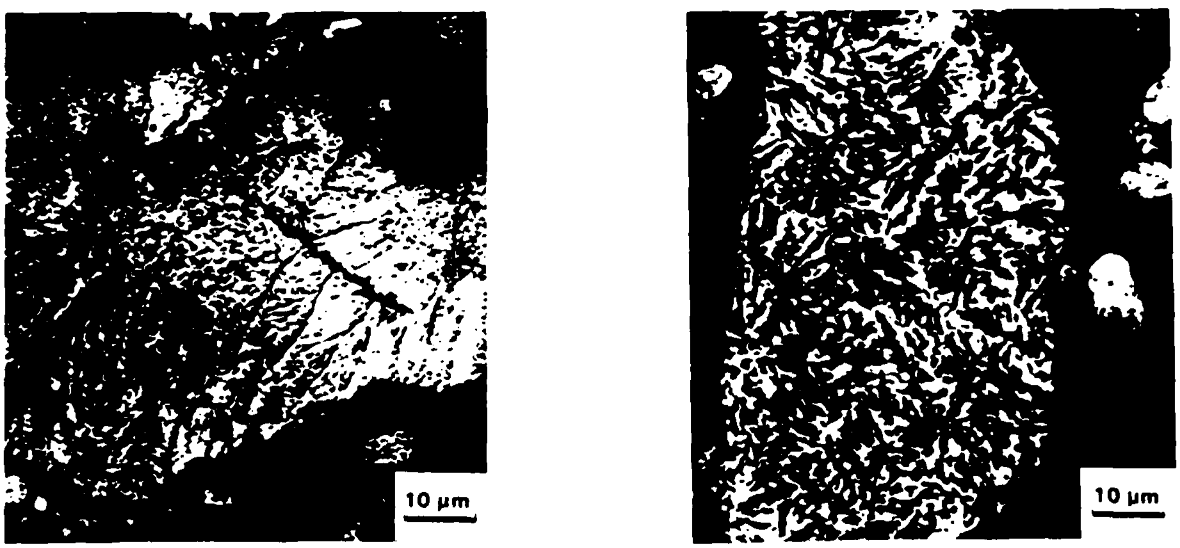



Figure 1: Air-atomized Al-Fe-Ni powder (SEM).




(a) (b)

Figure 2: Air-atomized Al-Fe-Ni powder; optical micrographs.
(a) Fine structure (A); (b) Coarse structure (B).




(a) (b)

Figure 3: Powder after exposure for 1 hour at 400°C: optical micrographs.
(a) Fine structure (A); (b) Coarse structure (B).

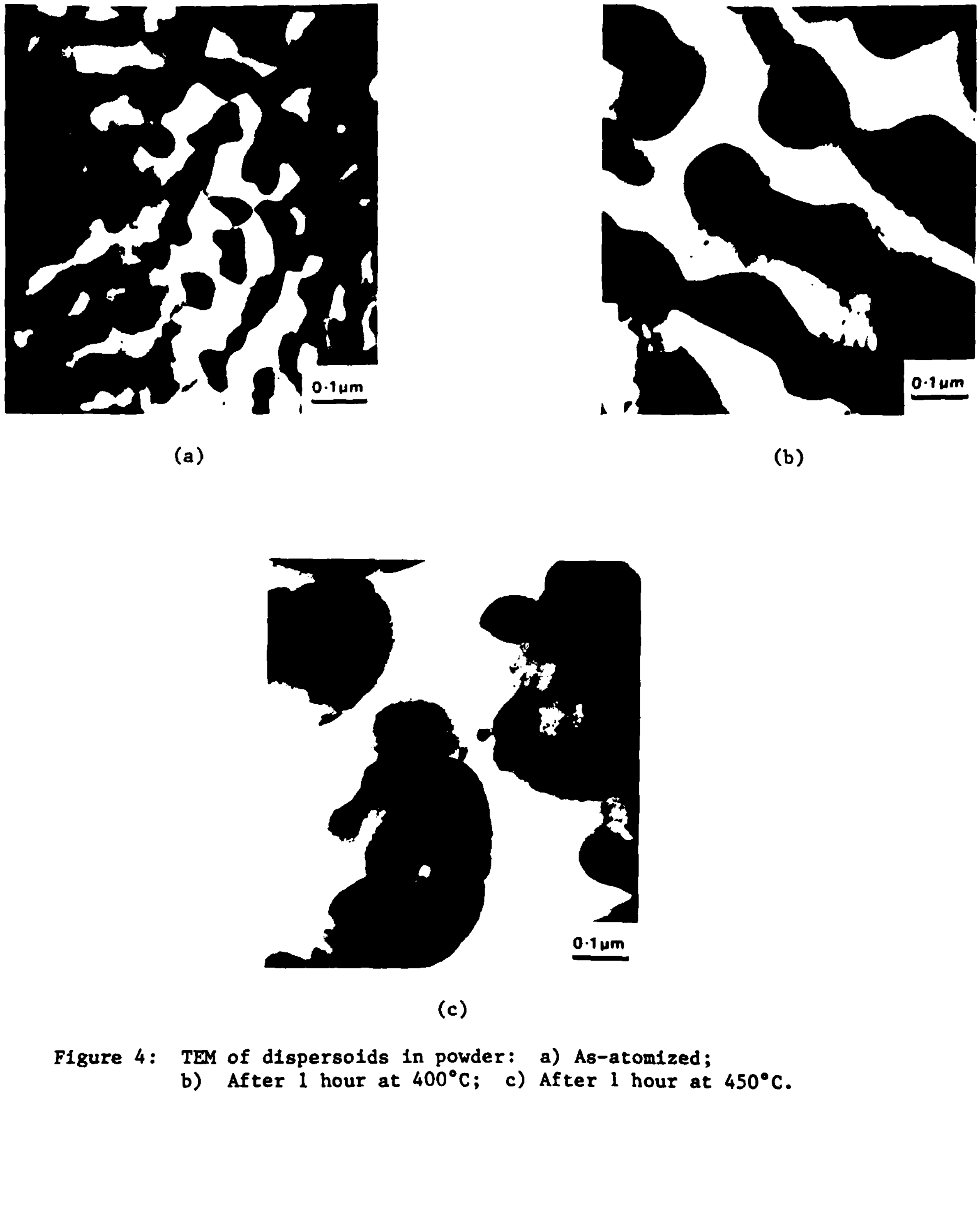



Figure 4: TEM of dispersoids in powder: a) As-atomized;
b) After 1 hour at 400°C; c) After 1 hour at 450°C.

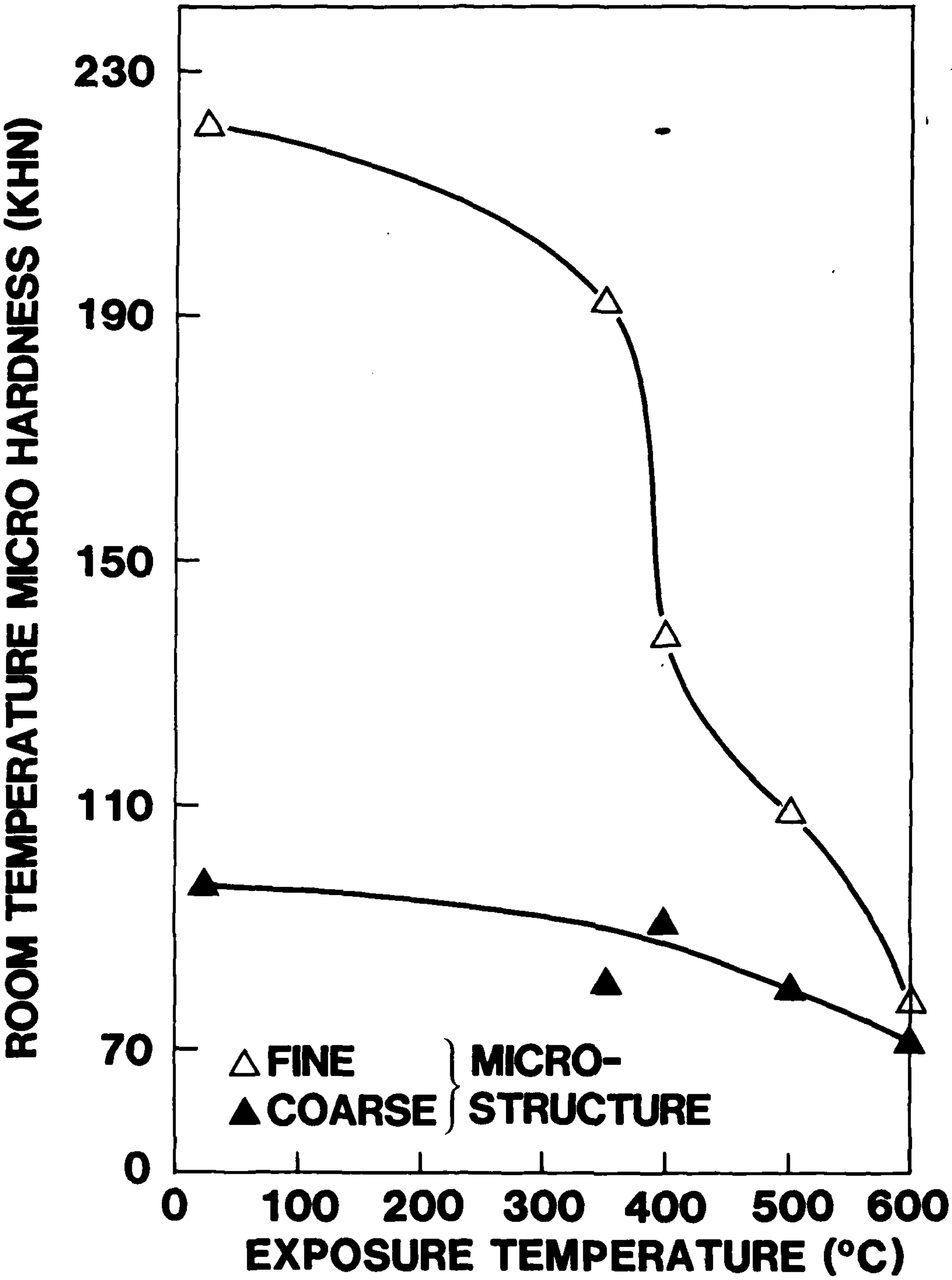
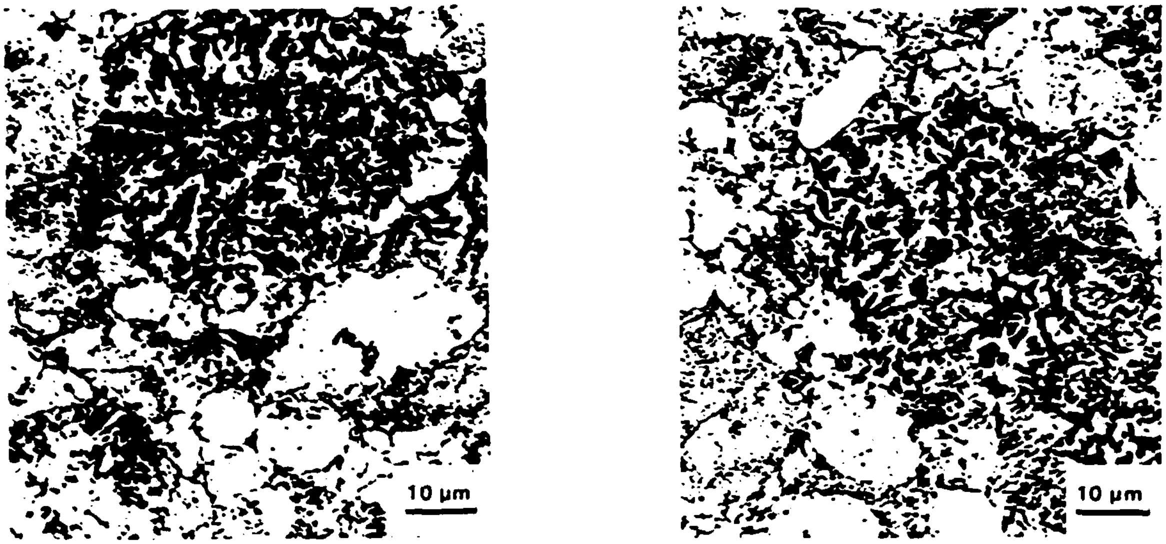
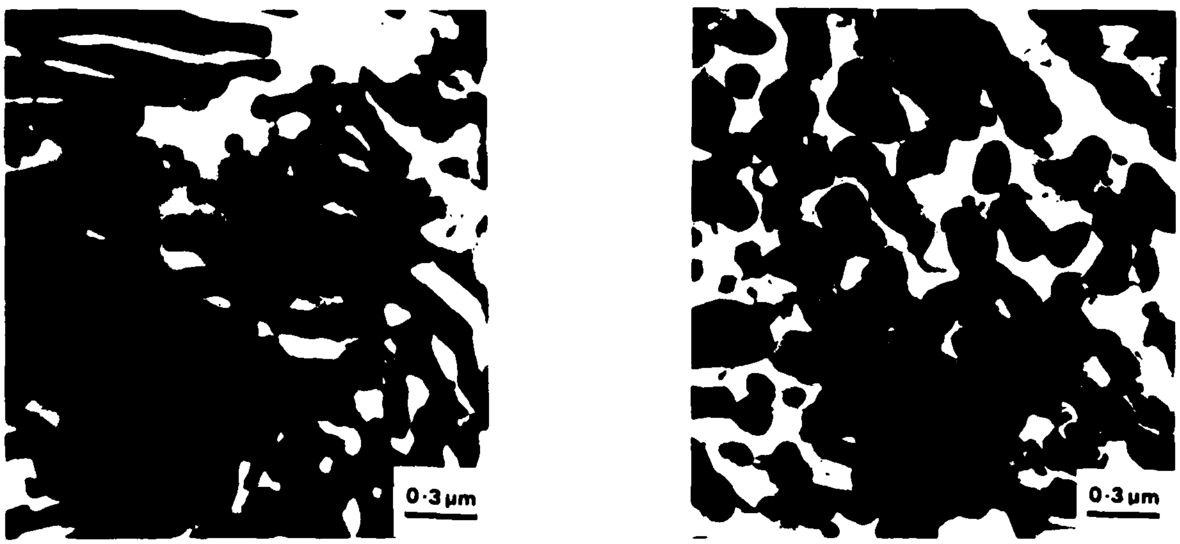



Figure 5: Microhardness of powder in fine and coarse regions, as a function of prior elevated temperature exposure (1 hour period).



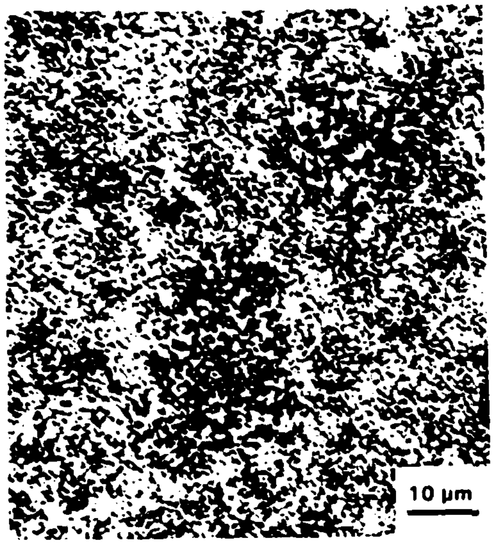

(a) (b)

Figure 6: Hot pressed powder; optical micrographs
(a) Hot pressed; (b) Hot pressed + 1 hour at 400°C.




(a) (b)

Figure 7: Hot pressed powder; TEM of fine-scale regions
(a) Hot pressed; (b) Hot pressed + 1 hour at 400°C.

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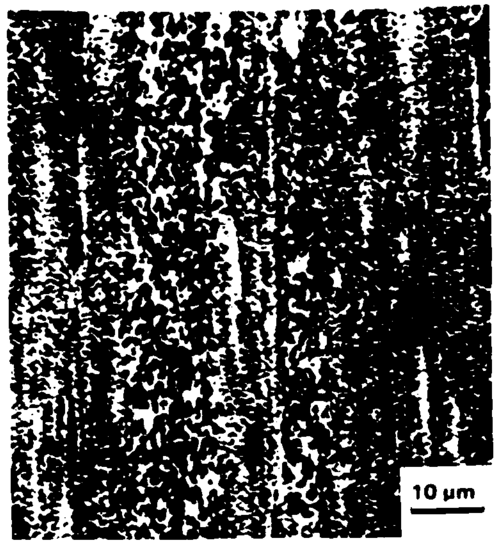


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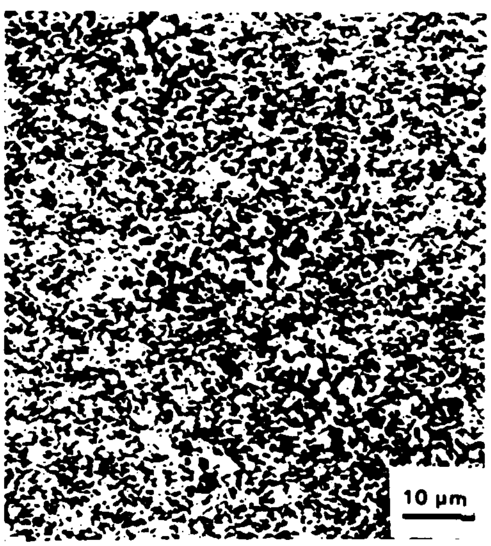


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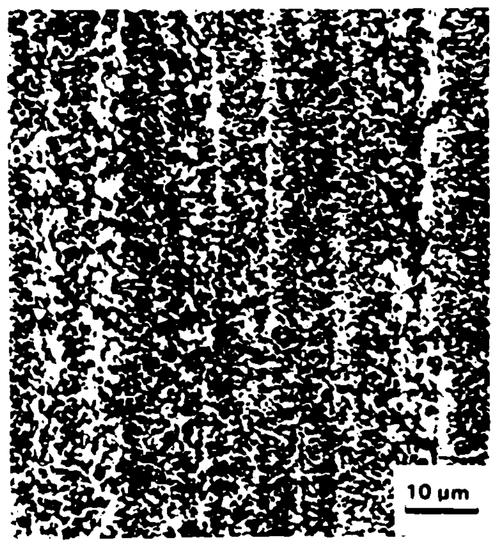
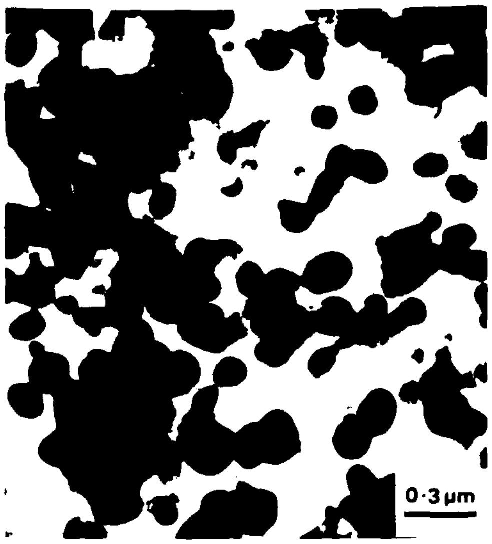



(d)

Figure 8: Optical micrographs of extruded powder. a) Transverse, as-extruded; b) Longitudinal, as-extruded; c) Transverse, after 1 hour at 400°C; d) Longitudinal after 1 hour at 400°C.

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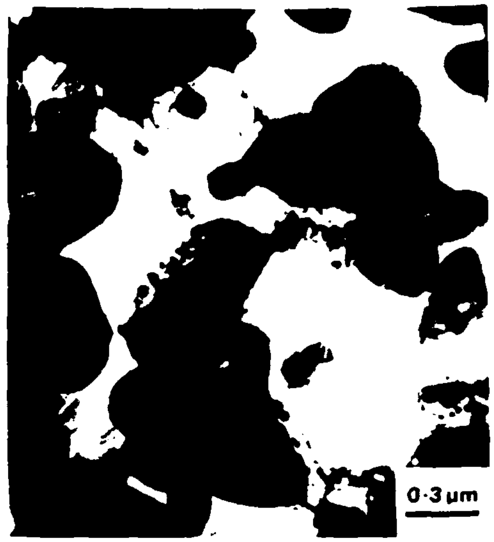


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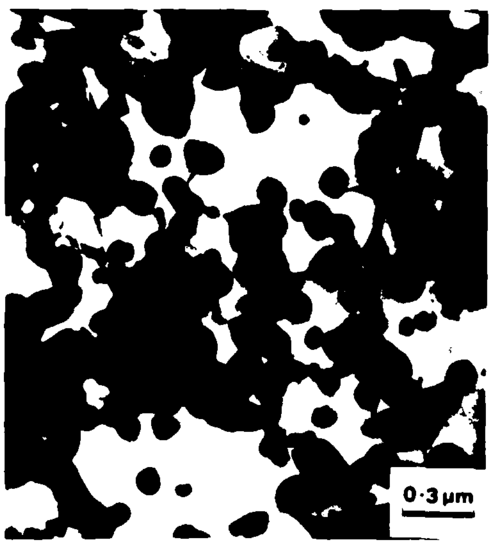


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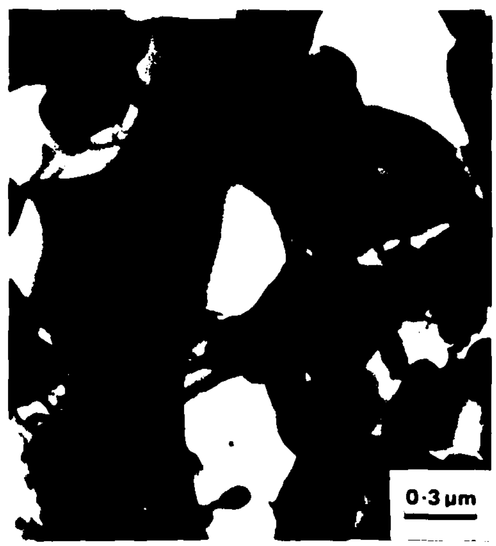


(d)

Figure 9: TEM of extruded material. a) Fine region, as-extruded; b) Coarse region, as-extruded; c) Fine region, after 1 hour at 400°C; d) Coarse region, after 1 hour at 400°C.

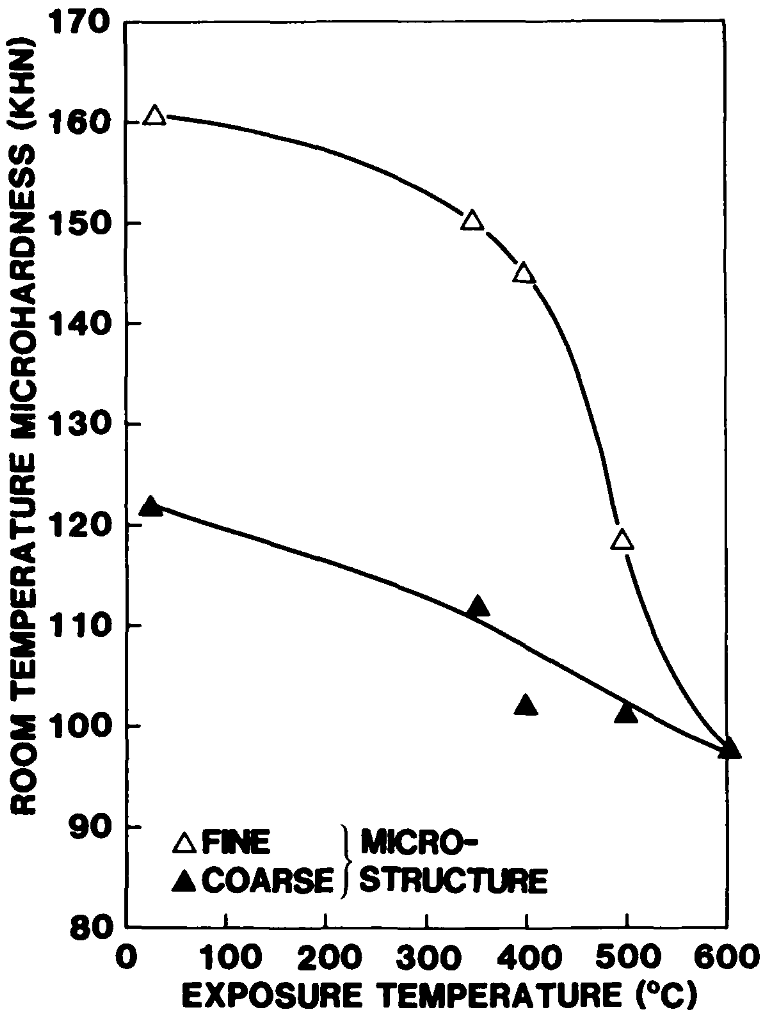


Figure 10: Microhardness-hot extruded powder in fine and coarse regions as a function of prior elevated temperature exposure (1 hour period).

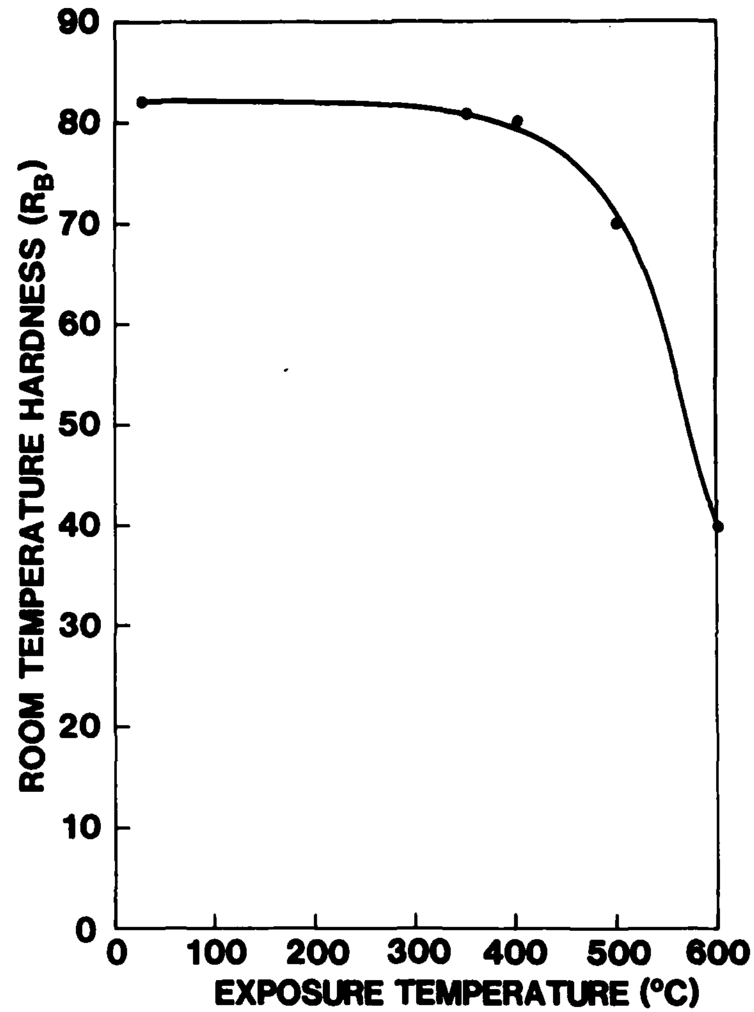


Figure 11: Macrohardness-hot extruded powder as a function of prior elevated temperature exposure (1 hour period).

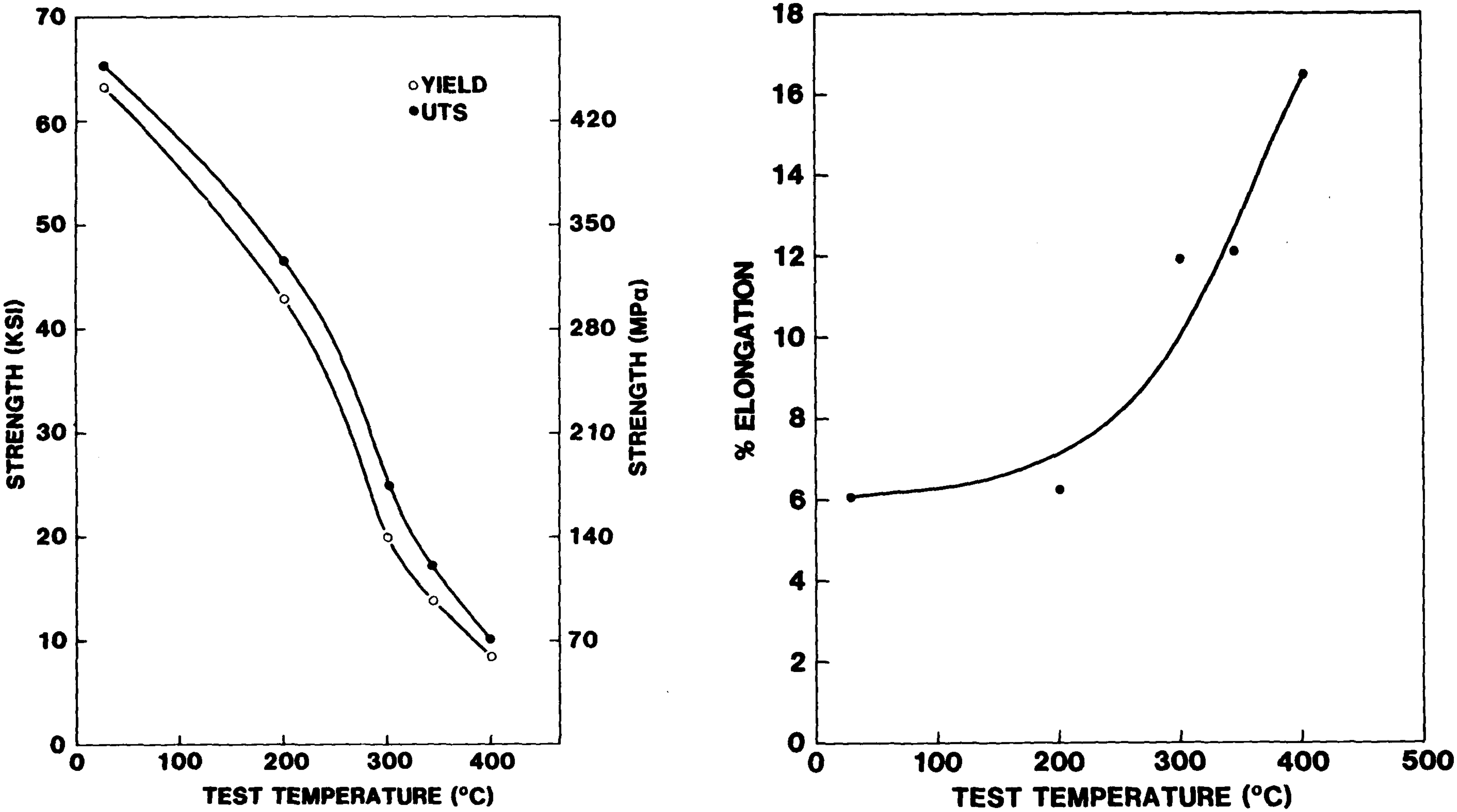


Figure 12: Hot tensile properties of extruded powder.

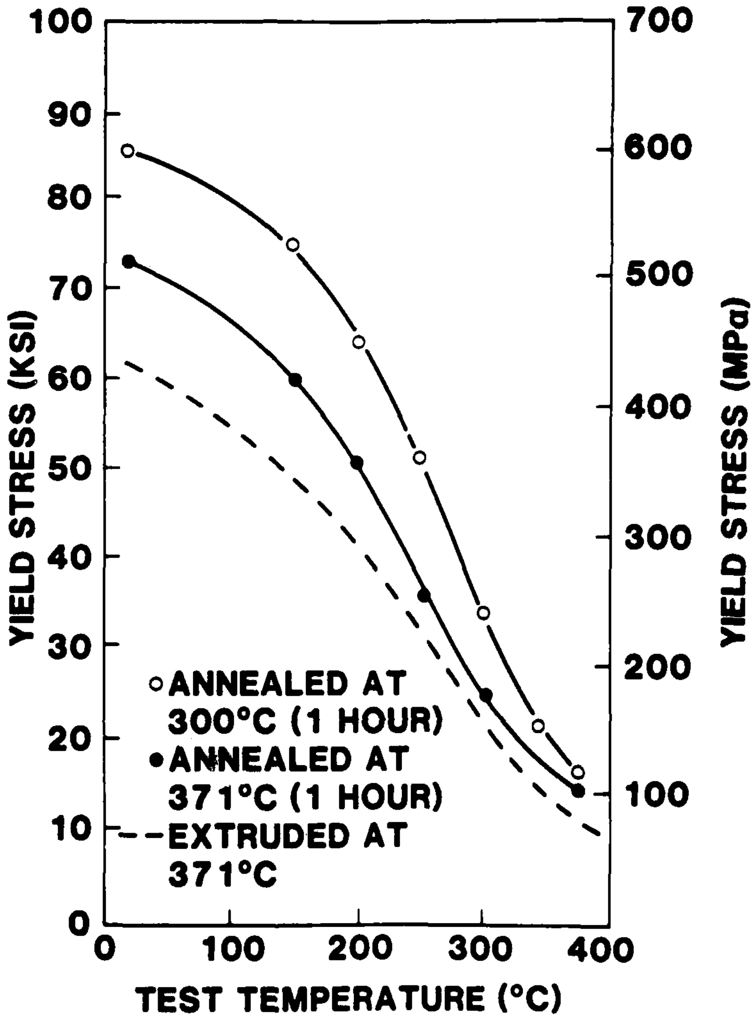


Figure 13: At-temperature yield strength of extruded and cold sintered/annealed powder.

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"Elevated Temperature Microstructural Stability and Properties of P/M Al-Fe-Ni Alloys", M. Premkumar, Ph.D. dissertation; in progress.

## PERSONNEL

A. Lawley - Professor and Co-Principal Investigator

M.J. Koczak - Professor and Co-Principal Investigator

E. Gutmanas - Visiting Professor

M. Premkumar - Ph.D. Student

COUPLING ACTIVITIES

a) Presentations - Lawley

"Powder Consolidation", ASM Seminar, Louisville, KY, October 1981.

"P/M Specialty Alloys", Beijing University of Iron and Steel Technology, People's Republic of China, November 1981.

"Fatigue of High-Strength Powder Metallurgy Aluminum Alloys", AIME Annual Meeting, Dallas, TX, February 1982.

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Presentations - Koczak

"Overview of Aluminum Powder Metallurgy Research", Sumitomo Light Metals, Nagoya, Japan, July 1982.

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"Powder Metallurgy Processing", Japan Institute of Metal Lecture , Kansai Area, University of Osaka, Osaka, Japan, September 1982.

"Fatigue in Aluminum Powder Metallurgy", Waseda University, Tokyo, Japan, September 1982.

b) <u>Technical Contacts with Other Laboratories</u>

Both principal investigators have interacted with other research personnel engaged in similar and related research in industry, government and academia. Contacts include:

| | | |
|---|---|---|
| Alcoa Technical Center | - | F.R. Billman, W.S. Cebulak, H.G. Paris, G.J. Hildeman |
| AFML/AFWAL | - | A.M. Adair, W.M. Griffith |
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